Physical properties of some zinc phosphate and polycarboxylate cements

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Several physical properties were measured for two zinc phosphate and three polycarboxylate cements. The specimens were tested in compression 24 h after they had been made. Two series of specimens were examined. In one series the cement powders were given a correct treatment, protected from atmospheric humidity. In the other series the powders were exposed for 1 week to ambient conditions with the temperature varying between 20° and 24°C and the relative humidity between 40% and 59%. The zinc phosphate cements were characterized by high values of modulus of elasticity and by plastic deformations less than 0.2%. The polycarboxylate cements were more flexible and also exhibited large plastic deformations. Compared with the zinc phosphate cements, therefore, the polycarboxylate cements had high values of resilience and toughness. The storing of cement powders exposed to atmospheric humidity for 1 week did not change the measured properties of the polycarboxylate cements. However, both strength and resilience were significantly reduced for one phosphate cement. ☐ *Dental cements; dental materials; physical properties*

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Polycarboxylate cements have been shown to be inferior to zinc phosphate cements with respect to compressive strength (1, 4, 9) and modulus of elasticity (9). The compressive strength of a zinc phosphate cement has been considered important for its retentive ability (1, 2). Reports have also been published, however, indicating that there is no correlation between the compressive strength of the cement and the retention of the restoration (1, 7). In one study pins cemented in pinholes in dentin were seen to be as well retained by a polycarboxylate cement as by a zinc phosphate cement (8). Clinical experience has also shown that polycarboxylate cements compare favorably with zinc phosphate cements when used to cement single crowns and bridges (3, 5, 6).

Since it seems that properties other than compressive strength, such as modulus of elasticity and elastic plastic behavior, may be indicative of the retentive ability of the luting medium, the aim of the present study was to characterize some zinc phosphate and polycarboxylate cements with respect to other mechanical properties in addition to compressive strength.

New polycarboxylate cements have been marketed which contain polyacrylic acid in the powder, so that the setting reaction starts when water is added. A pilot experiment indicated that such a cement powder was particularly subject to deterioration if exposed to an atmosphere of very high humidity. A comparison of the cements' response to exposure to air was therefore also made.

Materials and methods

The products listed in Table 1 were used in the study. The powder to liquid ratio for De Trey Zinc Cement Improved® was 2.4 g/ml. Phosphacap® is a capsulated product, so the powder to liquid ratio had to be the one given by the manufacturer.

The polycarboxylate cements were made using the powder to liquid ratios recommended by the manufacturers. In the case

Table 1. Products investigated

Code	Product	Cement type	Batch no.	Manufacturer
CAR	Carboxylon®	Polycarboxylate	Powder: 61602 Liquid: 61734	3M Co., USA
CER	Ceramco®	Polycarboxylate	98–765	Johnson & Johnson Dental Products Co., USA
DUR	Durelon®	Polycarboxylate	Powder: D 195 Liquid: G 274	Espe GmbH, FRG
DTZ	De Trey Zinc Cement Improved®	Zinc phosphate	Powder: YM5 80.02 Liquid: WJ 4YF	Amalgamated Dental, England
PHC	Phosphacap®	Zinc phosphate	Not indicated	Vivadent, Liechtenstein

of Ceramco® a reduced (80%) powder to liquid ratio was used in addition to the normal ratio, since this is a new product whose effects of variations in ratio do not seem to be known.

In the series of specimens to be studied with respect to an incorrect storage (all products except Phosphacap), the powder was arranged in a 2-mm-thick layer on a paper and left there for 1 week in a room in which the temperature varied between 20°C and 24°C and the relative humidity between 40% and 59%.

The specimens were made in accordance with ISO Standard 1566 for dental zinc phoshate cement with two exceptions: the split mold had a diameter of 6 mm and a height of 12 mm, and the mixing of the polycarboxylate cements was carried out as specified by the manufacturers.

The measuring of mechanical properties was done by the use of an Instron universal testing machine (Instron Model 1193) equipped with an extensometer (Instron Strain Gage Extensometer G–51–16 M). The machine was connected to a computer (ABC 80). Load and deformation data were sampled every 0.2 sec, and the following properties were calculated (Fig. 1): modulus of elasticity; stress and strain at fracture; compressive strength; 0.2% proof limit, stress, and strain; plastic strain; modulus of resilience; and toughness.

The mean and standard deviation values for each product/condition were based on the data from 10 specimens. The statistical significance of differences between means was evaluated by the use of Student's *t* test.

Results

The moduli of elasticity of the cements are presented in Fig. 2. It can be seen that the phosphate cements had a higher stiffness

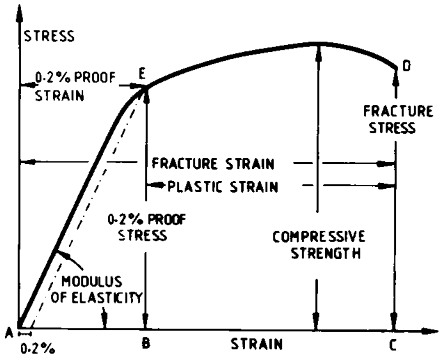

Fig. 1. Schematic presentation of the properties measured or calculated. Modulus of resilience: area ABEA; toughness: area ABCDEA.

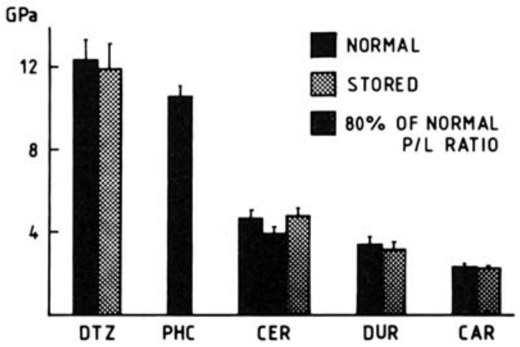

Fig. 2. Modulus of elasticity. The vertical bars on top of the columns represent one standard deviation.

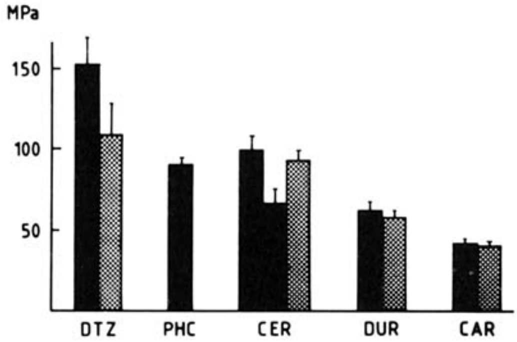

Fig. 3. Compressive strength. For explanation of column shades, see Fig. 2.

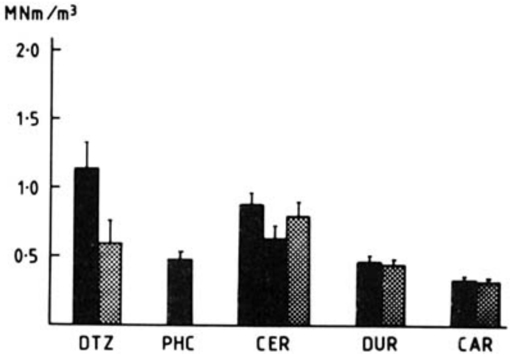

Fig. 5. Modulus of resilience. For explanation of column shades, see Fig. 2.

than the polycarboxylate cements. The effect of storing the powder in air for 1 week was negligible and in no case statistically discernible ($p > 0.15$). The reduced powder to liquid ratio in the case of Ceramco gave a decrease in modulus which was highly significant ($p < 0.001$).

The compressive strength data can be seen in Fig. 3. The strongest cement was De Trey Zinc Cement Improved. Ceramco exhibited a strength that was higher than that of any other carboxylate cement and also significantly higher than the strength of the phosphate cement Phosphacap ($p = 0.02$). The effect of incorrect storage was a slight and statistically not discernible decrease in strength ($p > 0.10$) for all cements except De Trey Zinc Cement Improved. In the case of the latter product the loss in strength was pronounced ($p < 0.001$).

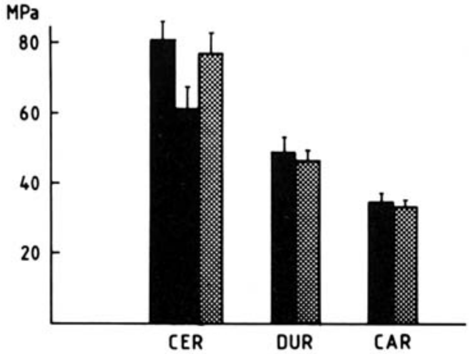

Fig. 4. 0.2% proof limit (stress). For explanation of column shades, see Fig. 2.

Fig. 4 illustrates the 0.2% proof limit for those products for which this property could be measured. The phosphate cements exhibited brittle fractures, and the plastic strain was less than 0.2% (compare Fig. 7). In all cases Ceramco had values of 0.2% proof limit which were considerably larger than those of the two other polycarboxylate cements. The reduction of the values resulting from the storing of the powder in air was not statistically discernible ($p > 0.12$). The low powder to liquid ratio used for Ceramco, on the other hand, implied a significant decrease ($p < 0.001$).

The moduli of resilience are presented in Fig. 5. De Trey Zinc Cement Improved had the highest value. The resilience of this product was markedly lowered when the powder had been exposed to air for 1 week ($p < 0.001$). Among the other four products the resilience of Ceramco was highest, even in the cases of a low powder to liquid ratio and 1 week's storage of the powder.

Since the phosphate cements exhibited a very low plastic strain (<0.2%), the toughness of these cements was equal to the resilience (Fig. 6). The polycarboxylate cements showed a large plastic deformation. The highest value was observed for Ceramco. The reduction in toughness encountered for powders stored in air was in no case statistically discernible ($p > 0.14$). The use of a low powder to liquid ratio for Ceramco gave a very pronounced decrease in toughness ($p < 0.001$).

Mean curves for the stress/strain relation-

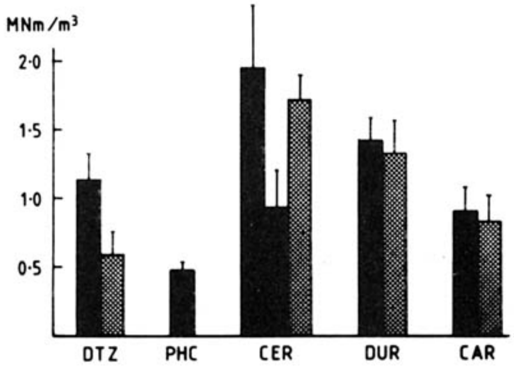

Fig. 6. Toughness. For explanation of column shades, see Fig. 2.

ship (Fig. 7) for all the cements were made on the basis of the stress and strain data collected. The graphs illustrate the characteristics of the various cement products: De Trey Zinc Cement Improved was seen to be stiff, strong, and brittle, and Phosphacap stiff, relatively weak, and brittle. The polycarboxylate cements were characterized by a high degree of plastic deformation. Among these cements Ceramco was outstanding in terms of strength and stiffness.

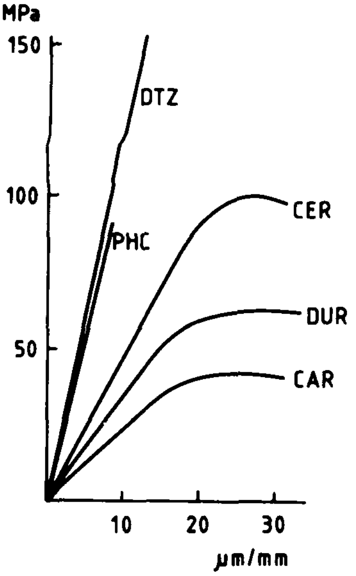

Fig. 7. Stress/strain graphs constructed from corresponding values of stress and strain.

Discussion

The present study has demonstrated for the products examined that polycarboxylate cements exhibit a lower stiffness and a greater plastic deformation than phosphate cements. This is a confirmation of findings made previously (9). The present study has also shown that the compressive strength of a polycarboxylate cement is not necessarily lower than the strength of phosphate cements. Ceramco is a polycarboxylate product that surpasses the phosphate cement Phosphacap with respect to compressive strength.

The favorable experience with polycarboxylate cements reported in the literature (3, 5, 6, 8) can hardly be accounted for by the compressive strength alone. As suggested earlier (9), the strain pattern of cements subjected to loading is probably also significant. It appears from the present results that the resilience of polycarboxylate cements matches or in some cases surpasses the resilience of phosphate cements. It can be imagined that having a high capacity of absorbing energy when loaded up to the elastic limit is an advantageous property for a luting cement. More information about this might be obtained if the present data could be correlated with results of retention studies.

When evaluating these results, it should be borne in mind that the stress and strain behavior of these cements may depend on the temperature at the time of testing. Thus, lower values of modulus of elasticity and strength and higher values of plastic strain were recorded when the testing was carried out at 37 °C (9). However, this effect is probably not so great as to invalidate the rather conspicuous differences seen between the products tested here.

Some detrimental effect of an incorrect storing of the cement powders before mixing was encountered. It was of interest, however, that the reduction in the various properties was most pronounced for De Trey Zinc Cement Improved and that Ceramco was not particularly sensitive to such a mistreatment. This seems to imply that even though this product may have its properties greatly reduced when exposed to extreme ambient conditions, there does not seem to be any risk of this happening under normal clinical routines. The results show, however, that the good properties obtained for this product are jeopardized if the cement is mixed with a lower than normal powder to liquid ratio. In this respect the cement behaves in a manner that is typical of all cements. Viscosity problems should ideally be handled not by adjusting the powder to liquid ratios recommended but by controlling factors such as the convergence angle of the preparation, temperature of slabs and spatulas, the fit of the crown, and in some cases by venting.

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